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An Elegant Low-cost Materials Solution for Achieving Low Insertion Loss, Affordable Tunable Filters for Next Generation Mobile Communications Platforms

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14. ABSTRACT

The effect ultraviolet (UV)-assisted annealing on the structural, dielectric, and insulating properties of barium strontium titanate (BST) thin films was investigated. The material properties of BST films, prepared by RF sputter deposition, annealed via conventional thermal annealing and UV-assisted thermal annealing process protocols were compared and evaluated. The x-ray diffraction results showed contracted lattice constants and enhanced crystallinity for the RF sputtered BST films annealed via the UV-assist treatment. Isothermal-temporal annealing experiments, over a broad range of annealing times ~12 to 225 min, revealed that the films annealed utilizing UV-assist possessed enhanced crystallization, and lattice parameters closer to bulk values with respect to the conventional thermal annealed films. The dielectric loss and leakage current density of the films, evaluated from 0 to 8V, was significantly reduced by employing UV-assisted annealing. The improved dielectric loss and leakage characteristics were attained without degradation of film tunability, hence material property balance was sustained. Mitigation of oxygen vacancies, associated with the UV-assisted annealing, is deemed responsible for the improvement of the dielectric and electrical properties of the BST thin films.

15. SUBJECT TERMS

Tunable devices, material design, complex oxide, UV photon irradiation

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1. Objective

The objective of this effort was to develop a novel materials technology solution to achieve high-Q perovskite oxide thin film materials to enable enhanced performance, low-cost tunable filters for next generation communications platforms.

2. Approach

For this fiscal year 2008 (FY08) Director's Research Initiative (DRI), we developed a post-growth ultraviolet (UV)-oxidation process science protocol to improve the quality factor (Q) and leakage characteristics (I_L) of complex multi-component dielectric films grown by practical industry standard, low-cost, large area, rapid deposition, and property uniform growth techniques. $Ba_{1-x}Sr_xTiO_3$, (BST) thin films were fabricated via a combinational processing technique which blends RF-sputtering and photon irradiation annealing to achieve balanced BST material properties, i.e., property enhancement without degradation of required performance metrics. This research introduced non-complex affordable processing routes for fabricating tunable active thin film oxides with balanced competing material property properties (dielectric loss and tunability) requirements.

3. Results and Discussion

Thin-film BST is a promising dielectric material for active elements in tunable devices such as filters, varactors, delay lines, phase shifters, and voltage controlled oscillators (*1–5*). Although the possibility of using BST thin-film varactors in tunable filters has been recognized for quite some time, this application space has not been fully exploited since most filter applications place a high premium on insertion-loss in the pass-band while maintaining high tunability. To date, the technology for BST fabrication has not been able to satisfy the required balance of these two material properties while still maintaining excellent reliability and device affordability. Conventional approaches for lowering thin-film BST dielectric loss include: acceptor doping (*6*), modification of film stress (*4, 7*), film texturing (*8*), ferroelectric-low loss oxide material layering (*9*), and improving the film/electrode interface quality by using refractory metals and/or adding buffer layers between the film and bottom electrode (*10, 11*). Although successful at lowering dielectric loss, unfortunately these approaches often lead to severe degradation of the films tunability and frequently involve complex non-industry standard film processing steps which, in turn, compromise device affordability. Thus, there is a critical need to develop new

material designs and/or film processing methods that enable maintenance of BST Q-factor (low dielectric loss/insertion loss) without compromising the material tunability (η~2:1) and other required filter performance metrics, such as device impedance matching (ε_r<500), film resistivity/conductivity (leakage current ~milliampere range) and long-term reliability (long-term performance under extended applied voltage and temperature).

The $Ba_{0.60}Sr_{0.40}TiO_3$ [BST (60/40)] thin films (~100 nm thickness) were RF sputtered from a stoichiometric $Ba_{0.60}Sr_{0.40}TiO_3$ ceramic target onto platinum silicide (PtSi) and *r*-plane sapphire substrates. The as-deposited films were annealed via conventional substrate thermal annealing and UV-assisted photon irradiation annealing. Both sets of films were in-situ annealed at variable temperatures (615–783 °C) and times (15–225 min) in a custom built load lock annealing chamber adjoining the sputtering chamber. The annealing chamber was designed with a substrate heater, precision oxygen partial pressure control, and equipped with *in situ* photon sources facing the sample for performing UV-assisted annealing. The UV photon source produces radiation between 185 and 254 nm and is operated at 50 mW/cm^2. In order to evaluate the effects of the UV-assisted annealing with respect to conventional *in situ* thermal annealing, the films were characterized for structural, dielectric, and insulating properties. The dielectric properties were measured in the metal-insulator-metal (MIM) device configuration using Pt top and bottom electrodes.

In order to minimize the effect of x-ray peak interference and evaluate/compare the structural properties of both the UV annealed and conventional thermally annealed BST films, glancing angle x-ray diffraction (GAXRD) was conducted using BST films grown on r-plane sapphire without electrode metallization. Specifically, the films were annealed for 60 min at temperatures ranging from 615 to 783 °C with and without UV treatment in an oxygen environment. The x-ray patterns of the conventionally annealed BST thin films and the films annealed with UV assist are shown in figure 1. The annealed films showed no evidence of secondary phase formation, as no peaks other than (100), (110), (111), (200), and (211) BST peaks were observed. It should be noted that for the conventional thermally annealed BST films, there was a slight shift of the x-ray peaks to lower angles (corresponding to larger interplanar spacing) with respect to that of the UV-assist annealed BST films. This downshift in the peak position is indicative of the fact that the lattice parameter of the conventionally thermal annealed BST films, based on the (110) x-ray peak, i.e., at 700 °C $a_{\text{conventional anneal}}$ = 4.01 Å as opposed to $a_{\text{UV-assisted anneal}}$ = 3.99 Å, was slightly larger than that for the film annealed with UV assist. To further detail this observed difference in lattice parameter between the two annealing methods, the experimental calculated lattice parameters, using the (110) x-ray peak, for both sets of films are shown as a function of variable annealing temperatures in figure 2. The dotted-dashed line in figure 2 represents the lattice parameter (*12*) for bulk ceramic $Ba_{0.60}Sr_{0.40}TiO_3$. It is suggested that the UV-treated films display structural characteristics closer to that of bulk BST, i.e., lattice parameter closer to bulk with respect to the conventionally annealed BST film, advocating a reduction in film stress resultant of defect (oxygen vacancies) annihilation in the film with respect to the non UV-treated films.

Oxygen vacancies affect the nearest neighbor distance by reducing the Coulomb attractive force between cations and anions, resulting in an increased lattice parameter (*13*). The oxygen vacancy site, being a prevalent defect in perovskite oxide films, has a net positive value thus repelling the barium (Ba), strontium (Sr), and titanium (Ti) atoms causing a distortion in the lattice parameter. It is proposed that the influx of activated oxygen during UV annealing allows for a greater compensation of the vacancies resulting in the reduction of the lattice parameter, and ultimately alleviating film strain by filling oxygen vacancies. Extrapolating the data, it is important to note that UV treatment can allow for the reduction in fabrication temperature by roughly 100–150 °C with respect to the non-UV treated films while maintaining the same degree of stoichiometry and crystallinity.

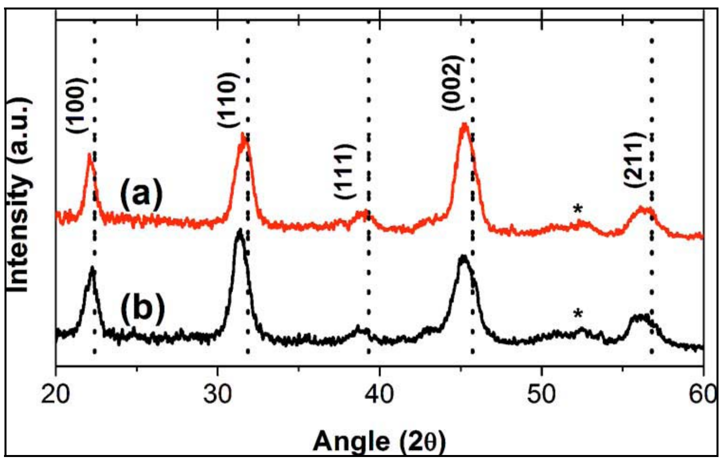

Figure 1. GAXRD patterns of BST thin films annealed at 700 °C for 60 min (a) with UV treatment and (b) without UV treatment. Dotted lines represent the BST (60/40) bulk peak position (*12*); the * denotes the (024) peak from sapphire substrate.

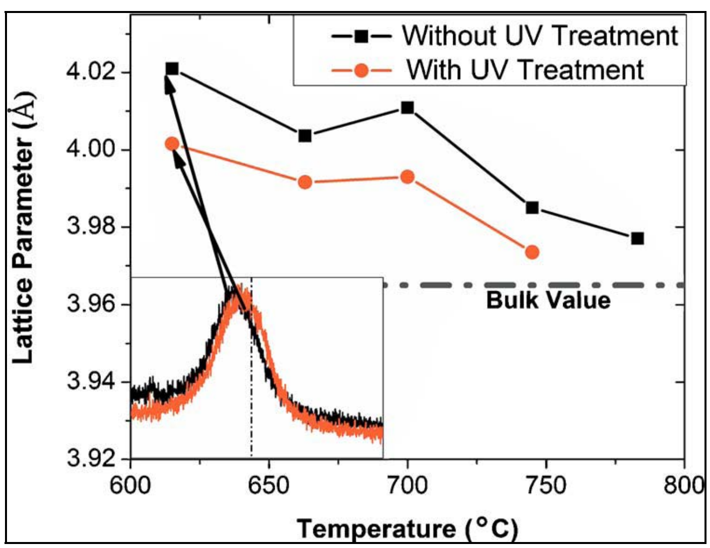

Figure 2. Change in lattice parameter as a function of annealing temperature for BST films annealed with (red filled circles) and without (black filled squares) UV-assist.

In order to determine the temporal effects of UV treatment on the BST films' structural quality, both sets of films were isothermally annealed at 700 °C as a function of variable annealing time from 15–225 min. Figures 3(a) and 3(b) show the change in full width at half maximum (FWHM) and lattice parameter of BST films annealed with and without UV assist, respectively. The steep decrease in FWHM, observed for both sets of films, as the time is increased from 15 to 30 min, suggests an increase in the grain size over a relatively short time interval. The decrease in FWHM as a function of annealing time is more pronounced for the UV treated film with respect to that of the conventionally annealed film. Notably, in both sets of films, the FWHM appears to approach a near-steady state after annealing for 75 min, i.e., additional annealing time does not drastically enhance the films grain size. Furthermore, the data in figure 3(a) (narrower FWHM of UV-assist treated film) demonstrates that for equivalent time and temperature conditions, the film annealed with UV assist is either more developed (possesses a higher degree of crystallinity/larger grain size) and/or possesses less residual stress, than the film annealed without UV-assist.

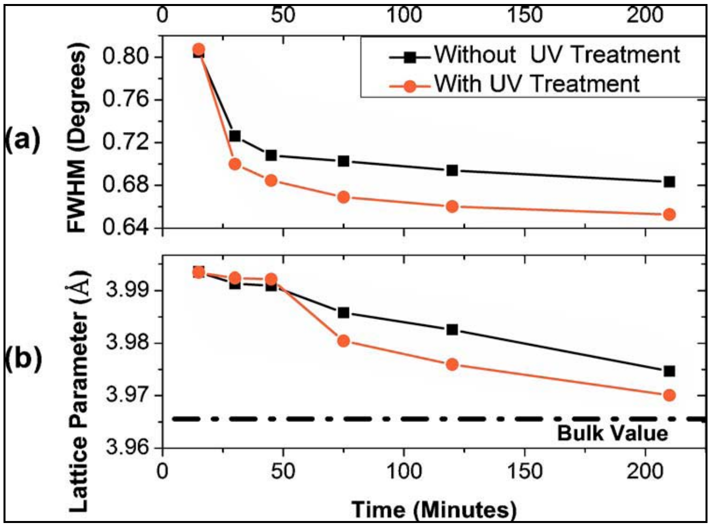

Figure 3. Variation in (a) FWHM and (b) lattice parameter of the 700 °C annealed UV assist (red filled circles) and non-UV treated (black filled squares) BST (60/40) films as a function of annealing time.

Figure 3(b) displays the variation in lattice parameter as a function of increasing annealing time for films annealed at 700 °C with and without UV assist. After annealing for ~45 min, there is a divergence in the lattice parameter such that at the same annealing time the lattice parameter of the UV-assist annealed film has contracted substantially with respect to that of the non-UV annealed film, and hence the lattice parameter is closer to that of bulk BST. This result suggests that UV irradiation has a dramatic effect on the film structure; whereby, the more pronounced

lattice parameter contraction observed for the UV treated films advocates that UV irradiation leads to a reduction in oxygen vacancies, hence yielding lower residual stress in the UV-annealed film.

Figure 4 displays the permittivity and tunability of the two sets of films as a function of applied bias. The permittivity of the UV annealed ($\varepsilon_{v=0}$=96) BST film is slightly lower than that of the non-UV annealed film annealed ($\varepsilon_{v=0}$= 108). This may be related to the lattice parameter difference in the two samples as the ionic displacement is reduced, lowering the net polarization. This decrease in polarization lowers the dielectric constant which can result in lower dielectric loss (*14*). The permittivity for both sets of films is reasonable (i.e., ε_r <500) for device impedance matching purposes (*15, 16*), thereby allowing efficient power transfer in the device. What is also important in figure 4 is the tunability data, whereby tunability (η) is defined as $\eta = \Delta C/C_0$, and ΔC is the change in capacitance relative to zero-bias capacitance C_0. Specifically, the tunability is virtually the same for both annealing treatments, with a maximum of ~35% at ±10 V applied bias. This tunability value is very reasonable for practical filter applications. Furthermore, the measured dielectric loss for both sets of films established that the dissipation factor for the UV-treated film was reduced by ~20% with respect to that of the non-UV treated film (*17*). This data is aligned with the lattice parameter findings (figure 2) and support the UV-assist's consequence to mitigate oxygen vacancies, thereby resulting in improved dielectric loss.

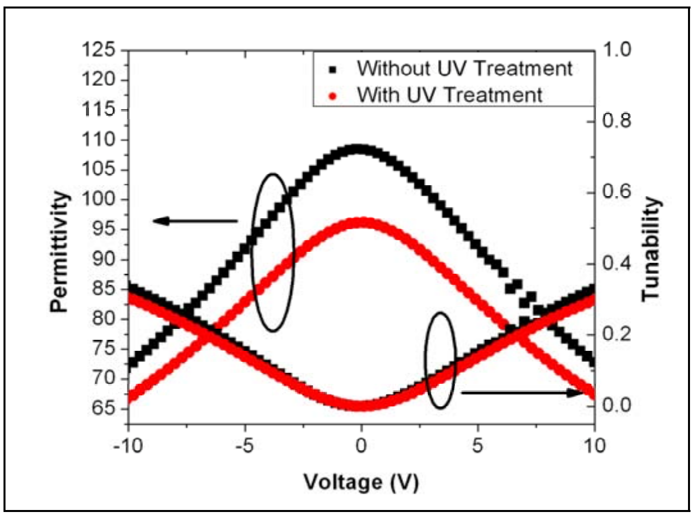

Figure 4. Dielectric response as a function of applied bias for the BST films annealed at 700 °C with (red filled circles) and without UV-assist (black filled squares).

Leakage current is one of the limiting factors for the suitability of a dielectric material for filter/tunable device applications (*14*). Large leakage currents arise from excess oxygen vacancies, since a higher concentration of oxygen point defects will give the BST *n*-type conductivity. These vacancies arise during deposition or post-deposition anneals due to the low molecular mass of the oxygen with respect to the other deposited atoms (*3*). It has been argued

that the major factors that influence leakage characteristics of BST films are oxygen vacancies generated during deposition, annealing of the BST film in low oxygen environments and during sputtering of the top electrode, and mechanical damage imposed by sputtering of the top electrode (*3*). The leakage current characteristics of BST thin films were measured using MIM capacitors. The relation of the leakage current density versus the applied bias is shown for both the UV-assist and conventionally thermal annealed BST films in figure 5. In contrast to conventional thermal annealing, the UV annealed BST film has a significantly lower leakage current density in reverse bias (bias voltage is defined as negative when a negative voltage is applied to the top Pt electrode). For example, the current density of the UV-assist annealed BST film is 7.73×10^{-7} A/cm^2 at −2.0 V applied bias and this value is significantly lower than that for the thermal annealed film (6.11×10^{-5} A/cm^2) for the same electric field. The oxygen vacancies generated at the top BST electrode by the sputtering process as well as the oxygen vacancies in the film from the initial sputtering conditions may act as electron trap sites, causing high leakage currents in the Pt/BST/PT capacitor structure. Since the UV radiation increases the influx of oxygen through the film, the vacancies are annihilated and thus the leakage current is decreased. Our results agree well with reported literature on the effect of oxygen annealing on leakage currents (*18*).

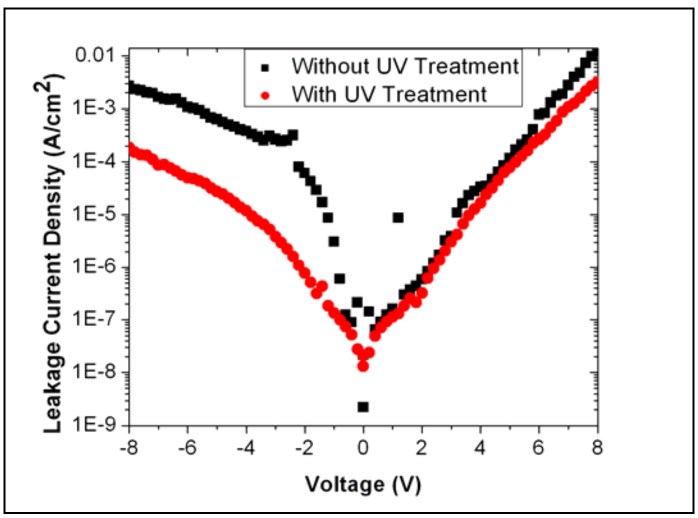

Figure 5. Variations of leakage current density with the applied reverse bias for the BST thin films annealed at 700 °C with (red filled circles) and without UV-assist (black filled squares).

4. Conclusions

This study demonstrates the potential for utilizing photon irradiation assisted annealing to achieve enhanced balanced material properties. Specifically, compared to conventional thermal annealed BST films, the UV-assist annealed films possessed improved leakage current

characteristics and dielectric loss with sustained tunabilities. The improved dielectric response, reduced leakage current and more pronounced lattice parameter contraction observed for the UV-treated films suggests that UV exposure plays a significant role in the annihilation of oxygen vacancies, which improves the film's material properties. Furthermore, the experimental data demonstrated that, for equivalent time and temperature annealing conditions, the UV-assist treated films possessed a higher degree of crystallinity compared to conventionally annealed films. This result suggests that UV-assisted annealing may enable reductions in synthesis temperature/time while facilitating improved material quality for tunable device applications.

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6. Transitions

1. Referred Journal Articles: 2

- Podpirka, A.; Cole, M. W.; Ramanathan, S. Effect of Photon Irradiation on Structural and Functional Properties of $Ba_xSr_{1-x}TiO_3$ (BST) Thin Films. *Appl. Phys. Lett.* **2008**, *92*, 212906.
- Cole, M. W.; Ngo, E.; Podpirka, A.; Ramanathan, S. Enhanced Performance Tunable Devices with Balanced Competing Material Properties. *Sensors*, submitted for publication, 2009.

2. Invited Presentations: 6

- Cole, M. W. *Advanced Multifunctional Materials for the Next Generation Microwave Communications Systems*, Physics Seminar Series, University of Texas, San Antonio, TX, November13, 2008.
- Cole, M. W.; Ngo, E; Podpirka, A; Ramanathan, S. *Oxygen Vacancy Mitigation with Application to Perovskite Oxide Thin Films*, Villa Conference on Complex Oxide Heterostructures, Orlando FL, Nov 2–6, 2008.
- Cole, M. W.; Ngo, E.; Podpirka, Adrian. *Comparison of the Material Properties of $Ba_{0.60}Sr_{0.40}TiO_3$ (BST) Thin Films Fabricated via UV-Assist and Conventional Thermal Process Science Protocols*, Symposium on Ferroelectrics & Multiferroics, MS&T Conference, Pittsburgh, PA, Oct 5–9, 2008.
- Podpirka, A.; Cole, M. W.; Ramanathan, S. *Manipulating Structural, Dielectric and insulating Properties of $BaSrTiO_3$ (BST) Thin Films by Ultra-violet Irradiation*, Symposium on Fabrication, Microstructures and Interfacial Properties of Multifunctional Oxide Thin Films, MS&T Conference, Pittsburgh, PA, Oct 5–9, 2008.
- Cole, M. W. *Novel Active Thin Films For Tunable Devices: Enhanced Dielectric Response While Maintaining Material Property Balance*, MSE Seminar Series, University of Florida, Gainesville, FL, Sept 12, 2008.
- Cole, M. W.; Ngo, E.;Podpirka, A.; Ramanathan, S. *$BaSrTiO_3$ Thin Films For Tunable Devices: Enhanced Dielectric Response While Maintaining Material Property Balance*, Symposium on Ferro-Electricity and Piezoelectricity, Intl. Materials Research Congress, Cancun, Mexico, August 16–23, 2008.

3. Tech Transfer of Experimental Results: 1

CERDEC, AMRDEC, and SOCOM: One patent disclosure in process.

List of Symbols, Abbreviations, and Acronyms

Ba	barium
BST	barium strontium titanate
DRI	Director's Research Initiative
FY08	fiscal year 2008
FWHM	full width at half maximum
GAXRD	glancing angle x-ray diffraction
MIM	metal-insulator-metal
PtSi	platinum silicide
Sr	strontium
Ti	titanium
UV	ultraviolet

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